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Abstract

We report on preliminary results of dynamic polarization at 2.5 Tesla and below 0.3 K of protons in a polyethylene foil covered by a superfluid ^4He film, providing the thermal contact to the mixing chamber of a dilution refrigerator. The results obtained let appear possible the development of ultrathin polarized solid targets, without solid walls and bulk liquid helium around, for particle scattering experiments.

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1. Introduction

Sizable nuclear polarizations have been obtained in powders and commercially available thin films (down to 20 μm thickness) of polyethylene, in which the free nitroxyl radical TEMPO¹ [1-4] had been introduced by diffusion. The advantages and the potential of ultrathin polarized polymer foils have also been indicated.

In a polarized solid target of conventional design [5] the target material is frequently placed in the mixing chamber of a dilution refrigerator. This design involves inevitably several layers of metal, plastic or quartz and liquid ³He/⁴He around the target, which cause many background events or even make low energy scattering experiments impossible.

To overcome to a large extent this inconvenience, we have investigated the possibility to polarize PE foils, covered only by a superfluid ⁴He film, which transports the heat from the material to be dynamically polarized to the mixing chamber. The results achieved so far in a test configuration are encouraging.

2. Preparation of the foils.

The PE foils were -as received- cut to strips and placed in a glass ampoule, sealable with a glass lid. This hollow lid was filled with the previously determined amount of TEMPO necessary to yield an optimum polarization [4]. Assuming a complete diffusion, the radical concentration corresponds to 2×10^{19} electron spins per gram of PE foil. The sealed ampoule was heated to 353 K and kept at this temperature during 24 hours. As a result, the radical disappeared from its original place and diffused into the polymer, which acquired a slightly orange colour.

The behaviour of the paramagnetic molecule in the polymeric medium was then studied by EPR. Neither pumping the foil during several hours, nor repeatedly cycling it between room temperature and 77 K, nor storing it in a glass tube at room temperature for more than a week had any effect on the intensity and shape of the EPR line. However, a marked decrease in

¹ 2,2,6,6-tetramethyl-piperidine-1-oxyl

TEMPO concentration was observed in probes, which had been for some time contained in a vessel machined off Stycast 1266. It seems that part of the nitroxyl radicals can migrate from their original location into the neighbouring material. This effect has led to some inconvenient consequences in our present experimental setup, described in more detail below.

3. Experimental details

The sample foil (0.02 g of 70 μm thick low density-PE foil, 12 mm $\times$ 18 mm), doped with TEMPO as described in the previous section, was mounted on a copper frame inside a small vacuum chamber made out of Stycast 1266 (Fig. 1), a material reasonably transparent to microwaves which can be easily machined and glued.

Thermal contact of the sample to the 1 mm thick copper frame (d) was ensured by clamping it with a copper counterpart in a sandwich construction kept together by two M1 screws. A 1 k Ω (Dale RCW550) RuO₂ thermometer (th) and a 350 Ω heater (h) were glued on top of the copper frame. The degree of proton polarization has been determined by means of a fast sweep Q-meter[6], for which a NMR coil of 0.8 mm brass wire with 1½ turns of 5 mm diameter was mounted as close as possible to the foil. Access to the vacuum chamber was provided by a conical plug, sealed with grease and tightened by a screw, all three parts (a,b and c) being made off Stycast 1266. The copper frame was glued into the plug using Stycast 2850GT, its bare copper end extending into the mixing chamber. Because the Stycast conducts almost no heat, the effective cooling power of the configuration is determined by the thermal conductance of the copper. A stainless steel capillary (e) was used to evacuate the chamber and to admit a controlled amount of helium in it.

The vacuum chamber was placed in a $\varnothing 18 \times 40$ mm brass container (the microwave „cavity“) and loaded in the mixing chamber of our standard polarized target test cryostat [7,8]. The magnetic field homogeneity was about 10^{-4} over the sample volume.

4. Experiments and Results

Our aim was to polarize dynamically the protons in a PE-foil kept in a closed cell, using only a superfluid ^4He film to assure the thermal contact to the mixing chamber of a dilution refrigerator. Different cooling conditions have been investigated in a series of consecutive experimental steps, gradually increasing the amount of ^4He gas condensed in the chamber, while permanently keeping the microwave irradiation at the same power level and its frequency locked to the previously determined optimum value.

Starting with the cell evacuated and the cooling of the foil provided only by the mechanical contact to the copper frame, +17% polarization could be achieved [curve (1) in Fig. 2]. The condensation of a small amount of ^4He gas, resulting in a coverage of all surfaces inside the chamber with a superfluid film of approximately $0.12\ \mu\text{m}$ thickness, led to a sudden drop in temperature, observable on the RuO_2 thermometer [(th) in Fig. 1], and to a corresponding steep increase in the polarization [curve (2) in Fig. 2]. After several hours, the same amount of ^4He was added again, but neither the temperature nor the polarization growth rate changed noticeably. Thereupon, the magnetic field was zeroed and the polarization process started all over again [curve (3) in Fig. 2], resulting in the same maximum polarization, namely +44%, achieved with the same growth rate. In order to assess the effectiveness of the „film cooling“, enough ^4He gas was then condensed to fill the whole cell: a slight temperature decrease could be noticed, and a somewhat faster polarization growth rate and a higher maximum polarization of +56% [curve (4) in Fig. 2] were measured. In our configuration the thick walls of the Stycast chamber, and not only the thin PE-foil, absorb microwaves, i.e. the ^4He film has also „to cool“ the inner side of the vacuum chamber. When the cell is completely filled with superfluid ^4He , the effective thermal resistance from the cell walls to the copper rod is reduced, which makes the „cooling“ more efficient. One can think that without microwave absorbing Stycast (and a larger heat exchange surface between superfluid film and mixing chamber), a thin superfluid film of ^4He could be sufficient to establish optimum polarizing conditions for a thin foil. One should remark that the temperature in the cell did not exceed 0.3 K, and was possibly even lower. At this temperature all the cooling takes place via the superfluid film, the contribution of the ^4He evaporation or the ^4He gas contact to the walls being negligible.

Further complications arose from the choice of Stycast for the construction of the vacuum chamber.

A lower degree of polarization, slower polarization growth rate, longer nuclear relaxation time and weaker EPR signals for PE foils which had remained enclosed in the Stycast cell at room temperature, as compared to the reference foils which had been kept in a glass vessel and a slight dynamic polarizability of the protons contained in Stycast, point out that TEMPO can migrate from a PE foil to Stycast even in absence of a physical contact. Judging from the strength and shape of the EPR line (Fig. 3), the radical concentration in the foil used in the described experiment (Fig. 2) was estimated to have become less than one half of the initial nominal 2×10^{19} electron spins per gram.

As mentioned in section 3, the degree of polarization was determined by means of a fast sweep Q-meter, comparing the absorptive part of the proton magnetic resonance signals after enhancement and at thermal equilibrium at 1.04 K and 2.17 K, assuming a Boltzmann distribution of independent spins for the hydrogen nuclei in the polyethylene molecules. The high proton content of the Stycast cell accounted for a large part of the thermal equilibrium NMR signal and thus made a precise calibration of the absolute polarization difficult. The polarization error is conservatively estimated to be 15 % relative and includes also the correction for the polarized background. The maximum polarization achieved in a foil placed inside the Stycast cell was +56% ($\Delta P/P = 15\%$) to be compared to +64 % and -58 % ($\Delta P/P = 5\%$) achieved in a piece of the same foil placed directly in the mixing chamber of the dilution refrigerator.

The lower polarization achieved in the case of the foil mounted in the Stycast chamber is most probably due to a loss of paramagnetic centers. This hypothesis is corroborated by the proton relaxation times measured at 2.5 Tesla, which increased from 37 s to 10 min at 2.17 K and from 519 s to 88 min at 1.04 K, in the foil mounted in the Stycast chamber.

5. Conclusions

Our measurements demonstrate that polymer foils, covered only by a thin film of superfluid ^4He at a temperature where the ^4He vapour pressure is less than 10^{-6} Torr, can be polarized rather well in a closed cell. The maximum polarization attainable in a carefully designed experimental environment is expected to be comparable to the case in which the foil is directly immersed in the liquid.

The successful test of this „cooling“ method can be considered as a first important step toward the realization of an ultrathin polarized solid target, which can be operated in a vacuum chamber. The technical realization of such a target depends critically upon the confinement of the superfluid ^4He film to the region of the mixing chamber, a problem which hopefully can be solved using film burners [9] and/or cesium coatings [10]. Such an instrument would allow a new class of measurements which could hardly be performed with a conventional polarized target.

Acknowledgement

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Figure Captions:

Figure 1:

Vacuum chamber consisting of parts a, b and c, with inside a copper frame (d) with heater (h) and thermometer (th). The upper half of the figure shows schematically where the PE-foil was situated and how it was mounted on the copper frame (d).

Figure 2:

A typical polarization experiment consisted of several consecutive steps. (1): polarize the foil in vacuum till saturation. (2): condense a small amount of ^4He to cover all surfaces with a superfluid film of $0.12\text{ }\mu\text{m}$ thickness and proceed to polarize. (3): admit again the same amount of gas to double the film thickness, and start the polarization process from the beginning. (4): fill the cell completely with liquid ^4He and start the polarization process all over again. During the whole experiment microwaves of fixed frequency (frequency locked) and power were fed into the cell.

Figure 3:

EPR spectrum of TEMPO diffused (during 24 hours at 353 K) into $70\text{ }\mu\text{m}$ thick PE foil. Thick line: Material with the nominal concentration of 2×10^{19} electron spins per gram, before loading into the vacuum chamber. Thin line: Same material after being mounted in the chamber and used for experiments. The radical concentration is in this case approximately 5×10^{18} electron spins per gram.

Figure 1

Figure 2

Figure 3